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Europäisches Patentamt
European Patent Office
Office européen des brevets

Publication number:

0 254 376
A1

EUROPEAN PATENT APPLICATION

(1) Application number: 87201426.1

(2) Int. Cl. C07H 13/06, B01F 17/00,
C11C 3/10

(3) Date of filing: 23.07.87

(4) Priority: 23.07.86 NL 8601904

(5) Date of publication of application:
27.01.88 Bulletin 88/04

(6) Designated Contracting States:
AT BE CH DE ES FR GB GR IT LI NL SE

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(13) Process for preparing partial polyol fatty acid esters.

- (14) A process for preparing partial polyol fatty acid esters, e.g. sucrose or sorbitol esters, is improved by
- (1) mixing the polyol with an alkaline catalyst such as KOH, NaOH or their carbonates, preferably in aqueous solution or dissolved in C₁₋₄ alcohols or ketones at atmospheric pressure and 10-80°C,
 - (2) preparing a mixture of fatty acid lower alkyl esters with an emulsifier, preferably a fatty acid soap, and
 - (3) adding the alkaline polyol solution to the mixture of (2), whereby preferably the solvent is removed during the addition of (1) to (2), e.g. at 60°C and 5 mbar.
- The reaction is improved so that partial polyol fatty acid esters with at most half of the polyol hydroxy groups being esterified are formed at 110-145°C within 1-4 hours.

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PROCESS FOR PREPARING PARTIAL POLYOL FATTY ACID ESTERS

The present invention relates to a process for the preparation of partial polyol fatty acid esters, in which one or more fatty acid lower alkyl esters are reacted with a polyol having at least 4 hydroxyl groups in the presence of an alkaline catalyst, and the partial polyol fatty acid esters formed are separated from the other reaction products and starting materials. In particular this is concerned with a new preparation of partial sucrose esters of fatty acids.

Further, the invention relates to the use of the partial polyol fatty acid esters thus prepared in animal feedstuffs, cosmetics, detergents, foodstuffs and pharmaceutical preparations.

In this specification, what is meant by "partial polyol fatty acid esters" are fatty acid esters of a polyol having at least 4 hydroxyl groups, of which not more than half of the hydroxyl groups have been esterified with fatty acid. For partial sucrose esters of fatty acids this means an average of at most 4 hydroxyl groups esterified with fatty acid.

These compounds are well known emulsifiers suitable for use in food and pharmaceutical products as well as in many other applications.

The most important group of polyols having at least 4 hydroxyl groups as described in literature about this subject is that of the sugar polyols, which group comprises the sugars, namely mono-, di- and trisaccharides, the corresponding sugar alcohols and derivatives thereof having at least 4 hydroxyl groups.

In literature many examples of sugar polyols are described, including the sugars lactose, maltose, raffinose and sucrose, the sugar alcohols erythritol, mannitol, sorbitol and xylitol and the sugar derivative alpha-methylglucoside (= alpha-methylether of glucose). The best known polyol fatty acid esters are the fatty acid esters of sucrose.

The aforementioned process is known. It is described, *inter alia*, in British patent specification GB 1,332,190 (Dai Ichi). For example, sucrose, fatty acid soap and water are mixed such that the sucrose is completely dissolved. To this solution a methyl ester of a fatty acid having 8-22 carbon atoms and a transesterification catalyst are added and the mixture is gradually heated under a gradually decreasing pressure until a substantially completely dehydrated melt is obtained without any substantial loss of fatty acid ester through hydrolysis, after which the resulting melt is kept at a temperature of 110-175°C to permit transesterification of the fatty acid methyl ester by the sucrose. Hydroxides, carbonates, bicarbonates, methoxides, ethoxides, and propoxides of potassium, sodium and lithium can be used as the transesterification catalyst.

According to this publication, the presence of soap in the aqueous sucrose solution is required to avoid aggregation of the powdery sucrose. The catalyst has to be added after the mixture of soap and sucrose has been prepared.

A serious disadvantage of this method is foam formation, which can occur during removal of the water, resulting in practical problems during application of this process on a technical scale.

Consequently there is still need of a relatively simple process for the preparation of partial polyol fatty acid esters with as little formation of by-products and troublesome foam formation as possible.

It has now been found that partial polyol fatty acid esters, in particular of sucrose, can be prepared in an elegant way if sucrose is first dissolved in aqueous KOH, separately a mixture of methyl esters and soaps is made, after which the alkaline sucrose solution is added to the mixture of soap/methyl ester under vacuum, during which at least part of the water is removed practically instantaneously. If desired, the soaps corresponding to the methyl esters used can be applied. The use of potassium soaps is preferred. After the mixture formed has been dried under vacuum, the mixture is heated to about 110-140°C to start formation of the partial sucrose fatty acid esters, which can be seen from the formation of methanol. When the reaction is completed or when either the sucrose or the methyl fatty acid is consumed totally, the formation of methanol will stop, but the reaction can be stopped any time. When the amount of methanol developed is measured, the point at which the reaction must be stopped for a specific degree of conversion can easily be determined.

Based on this finding and further experiments, the invention now provides a process for the preparation of partial polyol fatty acid esters, in which one or more fatty acid lower alkyl esters are reacted with an appropriate amount of a polyol having at least 4 hydroxyl groups in the presence of an alkaline catalyst and the partial polyol fatty acid esters formed are separated from the other reaction products and starting materials, which is characterized in that

(1) the polyol is mixed with the alkaline catalyst, forming a liquid system, if required using a non-toxic or easily removable solvent, in which system the alkaline catalyst may react with the polyol forming a catalytically active polyol anion, and

(2) this liquid system, optionally after any solvent used has first been removed, is combined with the fatty acid lower alkyl esters preferably containing an emulsifier, and the mixture thus formed is reacted under conditions such that the partial polyol fatty acid esters are formed.

In this specification polyol also comprises a partial fatty acid ester of a polyol having a low degree of substitution, which partial esters are intermediates in the conversion of polyol into polyol fatty acid esters having a higher degree of substitution.

Preferably a sugar polyol is used and particularly sucrose or sorbitol.

Suitable alkaline catalysts include the group consisting of alkali metals and alkaline earth metals, and the alkoxides, bicarbonates, carbonates, hydrides, hydroxides and alloys of these metals. KOH has been found to be particularly suitable as a cheap and effective alkaline catalyst, but also NaOH and the carbonates or bicarbonates of K or Na can be used with advantage. Although it can be argued whether the aforementioned KOH acts as a catalyst or as a reagent, which forms the actual catalyst or catalysts in the process, this description uses the term "catalyst", as does known literature about related reactions.

It is recommendable that in step (1) a solvent be used to improve the contact and, as a result, the reaction between polyol and alkaline catalyst. Suitable solvents include lower alcohols and/or ketones, e.g. a C₁₋₃-alcohol or -ketone. For sucrose as polyol and KOH as alkaline catalyst, water is a very suitable solvent.

Step (1) was carried out successfully at atmospheric pressure and room temperature. Partly owing to the solubility of sugar, other temperatures can be used as well, e.g. from 10-80°C or even from 40-70°C. Step (1) can also be carried out at a pressure higher or lower than atmospheric.

For practical purposes the alkaline catalyst of step (1) can be added in a molar ratio of catalyst:polyol of about (0.05 -1):1.

In principle many types of alkali-resistant emulsifiers can be used to improve contact of the ingredients in the reaction to be carried out in step (2). Known edible emulsifiers include mono/diglycerides, phosphatides, such as lecithin, and detergents such as soaps, sodium dodecyl sulphate and partial sugar esters of fatty acids.

In a particular embodiment of a process according to the invention, a fatty acid soap is incorporated as emulsifier in the fatty acid lower alkyl esters before the addition of the mixture containing the polyol and alkaline catalyst. The required soap can be made beforehand and be added in dry form. However, it is also quite possible that the fatty acid soap is formed in situ by partial saponification of the fatty acid lower alkyl esters or by neutralization of fatty acids added. In that case it is preferable to use a solvent in which an alkaline substance used for the saponification or neutralization can dissolve to improve the contact and, as a result, the saponification or neutralization. Suitable solvents include lower alcohols, preferably a C₁₋₃ alcohol, in particular methanol, and water. Other alkali-resistant solvents can also be used. When a solvent is used, it is recommendable that, after the in situ soap formation, the solvent be removed, e.g. by evaporation, before the resulting mixture containing fatty acid lower alkyl esters and soap is processed further in step (2). The amount of soap is preferably about 2-12 wt.% of the total reaction mixture.

Esters of lower alcohols, preferably of C₁₋₃ alcohols, are suitable for use as fatty acid lower alkyl esters. The fatty acids can be C₆₋₂₂ fatty acids, both saturated and unsaturated fatty acids. In the preparation of partial polyol fatty acid esters of C₆₋₁₂ fatty acids, starting from lower alkyl esters of C₆₋₁₂ fatty acids, there is a risk of part of the starting fatty acid lower alkyl esters evaporating; in that case, additional measures are to be taken to collect these starting materials and take them back into the reaction mixture.

It is advisable to use an excess of fatty acid lower alkyl esters and to control the degree of conversion by measuring the amount of lower alcohol developed. The excess of fatty acid lower alkyl esters can be removed during the refining of the reaction product according to known methods, e.g. by using the difference of HLB value between the fatty acid lower alkyl esters and the partial polyol fatty acid esters.

In an alternative procedure excess sucrose or other polyol is used, which excess can be removed during the refining because polyols are much more water-soluble than the partial polyol fatty acid esters.

The actual formation of partial polyol fatty acid esters takes place in step (2), which can be carried out at a temperature of about 100-180°C, preferably 110-145°C and at reduced pressure, in particular at about 1-50 mbar.

Although the scope of the invention is not restricted by theoretical statements, on the ground of data known from literature the following reactions can be indicated to illustrate the process according to the invention. It is assumed that in step (1), when sucrose and aqueous KOH are used, a sucrate anion is formed according to the equation



in which " \rightleftharpoons " means "in equilibrium with".

The relevant equilibrium is established practically instantaneously under the conditions used and is largely at the right side. In this connection it could be said that sucrose behaves like a weak acid (cf. J.A.Randleman, Jr.; Adv. in Carbohydr. Chem. 21 (1966), 209, 239-240, 244 and 246). For all that, when e.g. methyl esters are used, the water present in step (2) should be removed quickly, since the OH-ions still present may cause saponification of methyl esters with the aforesaid equilibrium shifting to the left at the expense of sucrate anion. On prolonged contact of sucrate anion with methyl esters in the presence of water, complete saponification takes place and no sucrate anion is left. When, after removal of the water, the temperature is raised to 110-145°C, this sucrate anion reacts with a methyl ester molecule according to generally accepted, recent theories on interesterification [cf. J.A.Heidal & P.C.Mork in (Proc.) 11th Scand. Symp. Lipids (1981), 147-152 (Puol. 1982)], in which a fatty acid chain is bound to sugar by means of an ester bond, releasing methoxide anion (CH_3O^-). This reacts immediately or simultaneously with a sugar molecule (partially or not partially acylated), with methanol being ultimately formed as visible reaction product.



This process is continued until all methyl esters have been used or all hydroxyl groups of sucrose have been acylated. Then, no methanol is produced any more and isolation of the partial polyol fatty acid esters can be proceeded to. By measuring the total amount of lower alcohol formed, e.g. methanol, the conversion can be determined, and when the desired degree of conversion is obtained, the reaction can be stopped by cooling.

Separation of the partial polyol fatty acid esters from the other reaction products and starting materials can be brought about by processes known per se, inter alia by using organic solvents, water and/or salting out.

In order to establish the degree of conversion, the amount of methanol released was determined.

Taking sucrose and methyl esters as starting products, after removal of the soap left after the reaction, the reaction product will consist of a mixture of methyl esters and sucrose esters of fatty acids. In a number of cases the hydroxyl number of this reaction product that had been freed of soap was determined, which is also a measure for the degree of conversion, whereby, of course, a correction needs to be made for the content of methyl esters in the reaction product. For example, the theoretical values of the hydroxyl number for sucrose fatty acid esters (SFAE) of groundnut oil fatty acids depends on the degree of conversion as follows:

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	SFAE (average number of esterified hydroxyl groups)	Hydroxyl number	Degree of conversion in %
40	8	0	100.0
45	7	26	87.5
	6	58	75.0
	5	101	62.5
50	4	160	50.0
	3	247	37.5
	2	386	25.0
55	1	647	12.5
	0	1310	0.0

Another indication of the conversion can be obtained by means of a mass balance for the sucrose esters. In most cases this balance will be in agreement with the conversion of methanol and/or the hydroxyl number found.

The process according to the invention further has, inter alia, the advantage that less foam is formed in the various steps of the process. It has been found that, especially when aqueous KOH is used instead of KOH/methanol, less foam occurs when soap is formed in situ. Likewise, when the water is removed quickly under vacuum, less foam is formed than when KOH/methanol is used.

The foaming problems mentioned do not arise, or to a far lesser degree, when using the process according to the invention, in particular when in step (2) aqueous KOH is used for forming soap in situ from the methyl esters or from the fatty acids and in step (1) for the formation of the liquid system containing polyol and alkaline catalyst.

The invention further relates to the partial polyol fatty acid esters prepared by a process according to the invention.

The invention also relates to a process for the preparation of animal feedstuffs, cosmetic preparations, detergent compositions, foodstuffs or pharmaceutical preparations, in which an effective amount of partial polyol fatty acid ester, which has been prepared according to the invention, is incorporated, e.g. as an emulsifier.

The invention is now illustrated by the following Examples without, however, being restricted thereto.

In Examples I-VIII as emulsifier soap was used which was prepared in situ. In Examples I and VIII this was done by partial saponification, in Examples II-VII by neutralization of fatty acids added. Although the Examples are directed to the preparation of sucrose fatty acid esters having at least 5 hydroxyl groups of the sucrose molecule esterified with fatty acid, it is clear from the degree of conversion given, that partial sucrose fatty acid esters having 1 to 4 hydroxyl groups esterified were present and can be isolated at lower degrees of conversion, e.g. about 12.5%, 25%, 37.5% and 50% conversion for an average fatty acid content of 1, 2, 3 and 4, respectively, per sucrose molecule

Example I

Step 1. Forming of the liquid system containing the sucrose and alkaline catalyst

25.4 g (74.3 mmol) sucrose and 0.896 g 85% KOH (13.6 mmol) were dissolved in 25 ml water at room temperature and at atmospheric pressure.

Step 2a. Forming of soap

8 g 85% KOH (0.12 mol) dissolved in 60 ml methanol was added to 125 g methyl ester of groundnut oil fatty acids (0.422 mol). The methanol was removed by boiling, followed by drying at 100-110°C and 1-2 mbar. Subsequently 225 g (0.760 mol) methyl ester of groundnut oil fatty acids was added.

Step 2b. Reaction

With vigorous stirring at 60°C and 2 mbar, the sucrose-containing alkaline solution of step (1) was added to the reaction product of step (2a), resulting in quick removal of the water during the addition. Thereafter, drying was carried out under the same conditions for about 30 minutes, resulting in a water content of less than 0.04%. Subsequently the temperature was raised to 120°C, after which sugar ester started to form. This could be observed by development of methanol, which was collected in a cold trap.

The molar ratio KOH:sucrose in step (1) was 0.183 and that of methyl ester:sucrose was 14.3. The amount of soap was 10.2%, calculated on the total reaction mixture.

The yield of methanol collected after reaction for 10 hours was 88% of the maximum amount of octa-ester calculated theoretically.

Example II

As in Example I, sucrose (25.4 g = 74.3 mmol) and 85% KOH (1.054 g = 16 mmol) were dissolved in water (25 ml).

- Subsequently a mixture of methyl esters of groundnut oil fatty acids (314 g = 1061 mmol) and fatty acids (32.67 g = 121 mmol of the product Pristerene 4911[®], ex Unichema, mainly consisting of saturated C₁₈ fatty acids) was prepared, to which a solution of 85% KOH (8 g = 121 mmol) in water (10 ml) was added at 60°C under vacuum.

Thereafter, the sucrose/sucrate solution was added to the soap dispersion in about 30 minutes at 60°C and 2 mbar. Further drying was then carried out for 30 minutes at 80°C and 2 mbar and the mixture was subsequently heated to 125°C, after which methanol started to develop. After reaction for 10 hours under these conditions, when 95% of the theoretical amount of methanol had been collected, calculated on complete conversion of the sucrose into octa-ester, the reaction was stopped by cooling to about 60°C.

During the 10 hour reaction period the total amount of methanol developed was determined every hour and the average degree of conversion was calculated. The values found were

Hours of	1	2	3	4	5	6	7	8	9	10
reaction										
conversion	6.7	12.7	22.5	45.6	71.1	81.3	86.7	90.4	92.9	95.4
average										
number of										
fatty acid	0.5	1.0	1.8	3.6	5.7	6.5	6.9	7.2	7.4	7.6
per sucrose										
molecule										
Partial sucrose					Sucrose fatty acid					
fatty acid esters					polyesters					

Thus, if the reaction was stopped during the first four hours, the majority of the sucrose fatty acid esters were partial esters, which could have been isolated.

After further processing the reaction mixture according to the BOSSIER method with 2-propanol/water in order to remove the soap, a hydroxyl number of 9.8 was measured.

The molar ratio KOH:sucrose in step (1) was 0.215 and the molar ratio methyl ester:sucrose was 14.3. The weight percentage of soap in the total mixture was 10.2.

Example III

Example II was repeated, except that the product Pristerene 4941[®], ex Unichema (36.8 g = 121 mmol, mainly consisting of saturated C₁₈₋₂₂ fatty acids) was now used as fatty acid source. The reaction was carried out at 140°C instead of 125°C. After 6 hours, 99% of the theoretical amount of methanol had been collected. The weight percentage of soap, calculated on the total mixture, was 10.9. The hydroxyl number before steaming but after washing with a mixture of 2-propanol/water for removal of the soap and after drying was 4.

As in Example II, the total amount of methanol developed during the reaction was determined every hour, giving the following values :

30 Thus the partial sucrose fatty acid esters were formed within about 1.75 hours.

Example II was repeated, except that the product Prifac 7960^a, ex Unichema (containing about 60% linoleic acid and about 22% oleic acid) was used as fatty acid source. After reaction for 9 hours, 95% of the theoretical amount of methanol had been collected.

As in Example II, measurement of the total amount of methanol development gave the following conversion :

Hours of	1	2	3	4	5	6	7	8	9	10
reaction										
conversion	14.9	57.1	76.4	82.6	85.5	89.7	92.3	94.3	95.0	95.8
average										
number of										
fatty acid	1.2	4.6	6.1	6.6	6.8	7.2	7.4	7.5	7.6	7.7
per sucrose										
molecule										
Partial sucrose	Sucrose fatty acid									
fatty acid	polyesters									
esters										

Thus the partial sucrose fatty acid esters were formed within about 1.75 hours.

Example V

Example II was repeated, except that methyl esters of fatty acids of soybean oil were used, hardened to 89°C (314 g = 1061 mmol) and, as fatty acid source the product Priolene 6930[®], ex Unichema (mainly consisting of oleic acid; 33 g = 121 mmol) 10 wt.% soap was used, calculated on the total mixture. After reaction for 9 hours, 96% of the theoretical amount of methanol had been collected.

As in Example II, measurement of the total amount of methanol developed gave the following degree of conversion, showing that within about 2.5 hours the partial sucrose fatty acid esters were formed.

Hours of	1	2	3	4	5	6	7	8	9
reaction									
conversion	10.5	25.4	61.3	80.9	87.1	90.2	92.7	95.1	96.3
average									
number of									
fatty acid	0.8	2.0	4.9	6.5	7.0	7.2	7.4	7.6	7.7
per sucrose									
molecule									
Partial sucrose	Sucrose fatty acid								
fatty acid esters	polyesters								

Example VI

- Example V was repeated, except that methyl esters of coconut oil fatty acids (244 g = 106 mmol) were used and the reaction was carried out at 110-118°C. 12.5% soap was used, calculated on the total mixture.
- After 10.5 hours, no methanol was formed any more, after which the reaction mixture was cooled and further processed. If reaction is stopped after about 1-2 hours, mainly partial sucrose fatty acid esters are formed.

10 Example VII

- As in Example I, sucrose (254 g = 0.743 mol) and 85% KOH (10.54 g = 0.16 mol) were dissolved in 250 ml water.
- Subsequently a solution of 85% KOH (80 g = 1.21 mol) in 100 ml water was added to methyl esters of soybean oil fatty acids (3500 g = 11.82 mol) at 70°C under 1 atm. nitrogen. After heating for 1.5 hours at 95°C, water and methanol were removed under vacuum. The sucrose/sucrate solution was then added to the soap dispersion at 60°C and 2 mbar. After drying under the same conditions, the mixture was heated to 125°C in order to start the reaction; 12 hours later, 99% of the theoretical amount of methanol had been collected.
- The conversion was followed as in Example II, showing that after 1.5 hours the sucrose was converted mainly into partial sucrose fatty acid esters.

Hours of reaction	1.5	2.5	4.5	7.5	12.0
conversion	42	73	86	96	99
average number of fatty acid per sucrose molecule	3.4	5.8	6.9	7.7	7.9

Example VIII

- A sucrose/sucrate solution was prepared as indicated in Example II.
- Subsequently 22.0 g of the product Pristerene 4941[®], ex Unichema (= 72 mmol) was added to methyl esters of groundnut oil fatty acids (314 g = 1061 mmol) at room temperature, which mixture was then neutralized by the addition of 85% KOH solution (4.94 g = 75 mmol in 6 ml water) at 60°C under vacuum.
- Thereafter, the sucrose/sucrate solution was added to the soap dispersion as in Example II. After drying for 30 minutes under the same conditions, the mixture was heated to 125°C in order to start the reaction; about 5 hours later, 100% of the theoretical amount of methanol had already been collected.
- The time, conversion and average number of fatty acid per sucrose molecule, showing that partial sucrose fatty acid esters were formed during the first 2 hours of reaction, were :

	Minutes of	105	165	225	285
5	reaction				
	%				
10	conversion	25	68	97	100
	average				
	number of				
15	fatty acid	2.0	6.2	7.8	8.0
	per sucrose				
	molecule				

20 The molar ratio KOH:sucrose in step (1) and methyl ester:sucrose was the same as in Example II. The amount of soap was 6.8 wt.%, calculated on the total mixture.

Claims

- 25 1. Process for the preparation of partial polyol fatty acid esters, in which one or more fatty acid lower alkyl esters are reacted with an appropriate amount of a polyol having at least 4 hydroxyl groups in the presence of an alkaline catalyst and the partial polyol fatty acid esters formed are separated from the other reaction products and starting materials, characterized in that
- 30 (1) the polyol is mixed with the alkaline catalyst, forming a liquid system, if required using a non-toxic or easily removable solvent, in which system the alkaline catalyst may react with the polyol forming a catalytically active polyol anion, and
- (2) this liquid system, optionally after any solvent used has first been removed, is combined with the fatty acid lower alkyl esters preferably containing an emulsifier, and the mixture thus formed is reacted
- 35 under conditions such that the partial polyol fatty acid esters are formed.
2. Process according to Claim 1, wherein a sugar polyol, preferably sucrose or sorbitol, is used as polyol
3. Process according to Claim 1, wherein the alkaline catalyst is selected from the group consisting of alkali metals and alkaline earth metals, and the alkoxides, carbonates, bicarbonates, hydrides, hydroxides and alloys of these metals, and preferably KOH is used as alkaline catalyst.
- 40 4. Process according to Claim 1, wherein step (1) is carried out at atmospheric pressure and at a temperature of about 10-80°C, and preferably a solvent is used selected from the group consisting of C₁₋₄ alcohols, C₁₋₄ ketones and water, and/or the alkaline catalyst is added in an amount such that the molar ratio of catalyst to polyol is in the range of about (0.05-1):1.
- 45 5. Process according to Claim 1, wherein in step (2) a fatty acid soap preferably in an amount of about 2-12 wt.% of the total reaction mixture is incorporated as emulsifier in the fatty acid lower alkyl esters, which fatty acid soap is preferably formed in situ either by partial saponification of the fatty acid esters or by neutralization of fatty acids added, whereby more preferably during the in situ formation of soap a solvent is used in which an alkaline substance used for the saponification or neutralization can dissolve, which solvent is preferably selected from the group consisting of C₁₋₄ alcohols and water and after the in situ soap formation, the solvent used can be removed by evaporation before the resulting mixture containing fatty acid lower alkyl esters and soap is further processed in step (2).
- 50 6. Process according to Claim 1, wherein esters of C₈₋₂₂-fatty acids and C₁₋₄ alcohols are used as fatty acid lower alkyl esters.
7. Process according to Claim 1, wherein step (2) is carried out at a temperature of about 100-180°C, preferably 110-145°C and a reduced pressure in particular of about 1-50 mbar.

8. Process for the preparation of an animal feedstuff, a cosmetic preparation, a detergent composition, a foodstuff or a pharmaceutical preparation, in which an effective amount of partial polyol fatty acid esters prepared according to any of Claims 1-7 is incorporated as an emulsifier

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EP 87 20 1426

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
A	EP-A-0 132 293 (THE PROCTER & GAMBLE CO.) * Claims *	1	C 07 H 13/06 B 01 F 17/00 C 11 C 3/10
A	EP-A-0 132 941 (THE PROCTER & GAMBLE CO.) * Claims *	1	
A	DE-A-2 051 766 (DAI-ICHI KOGYO SEIYAKU CO.) * Pages 23-25 *	1	

			TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
			C 07 H 13/00
The present search report has been drawn up for all claims			

Place of search THE HAGUE	Date of completion of the search 08-10-1987	Examiner VERHULST W.
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